HYDROPHOBIC SILICA

Reference to a Related Application

This application claims the benefit of provisional application 60/ 171,667 filed December 27, 1999 which is relied on and incorporated by reference.

Introduction and Background

This invention relates to a hydrophobic, pyrogenically produced silica, to a process for the production thereof and to the use thereof.

It is known to compact hydrophilic, pyrogenically produced silica (EP 0 280 854 B1). Disadvantageously, as tamped or bulk density increases, thickening action declines in a linear manner. Dispersibility also falls as density increases. This results in unwanted speckling. Thus, once compacted, a hydrophilic, pyrogenically produced silica may only be used for a limited number of applications.

It is therefore an object of the present invention to avoid the problems of compacted, hydrophobic, pyrogenically produced silica of the past.

Summary of the Invention

The above and other objects of the present invention can be achieved by developing a hydrophobic, pyrogenically produced silica having a tamped density of 55 to 200 g/l.

The tamped density is preferably from 60 to 200 g/l.

A feature of the present invention is a process for the production of the hydrophobic, pyrogenically produced silica having a bulk density of 55 to 200 g/l, which process is characterised in that pyrogenically produced silica is hydrophobized using known methods and then compacted.

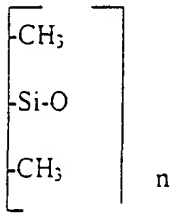
Hydrophobing can preferably be performed by means of halogen-free silanes. The chloride content of the silica can be less than or equal to 100 ppm, preferably from 10 to 100 ppm.

Compaction can be performed by means of a roller compactor. Compaction can preferably be performed by means of a belt filter press according to EP 0 280 851 B1, which reference is relied on and incorporated by reference.

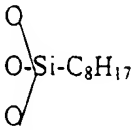
The hydrophobic, pyrogenically produced silica used for purposes of the present invention can be, for example, the silicas known as:

Aerosil R 812 or Aerosil R 812S, having the group -0-Si $(CH_3)_3$

Aerosil R 202, Aerosil MS 202, Aerosil MS 202, Aerosil R 106 or Aerosil R 104 having the group



Aerosil R 805 having the group



These are commercially available products from Degussa Hüls AG.

The hydrophobic, pyrogenic silica according to the invention having a tamped density of 55 to 200 g/l exhibits the following advantages:

Transport costs are distinctly lower as a result of the higher tamped density.

Once dispersed, the silica according to the invention is in the form of relatively small aggregates. In other words, the dispersions are more finely divided because the silica according to the invention is more readily dispersible.

The dispersions produced using the silica according to the invention exhibit a lower Grindometer value.

Both UV transmission transparency and visual transparency of the dispersions are distinctly improved by using the silica according to the invention.

Dispersions containing the silicas according to the invention exhibit distinctly increased stability because the tendency towards settling is distinctly lower.

Another advantage of the silica according to the invention is reduced dusting during incorporation and the distinctly reduced incorporation or wetting time in, for example, liquid systems.

In comparison with hydrophobic, pyrogenic silica of a lower bulk density, the hydrophobicity of the silica according to the invention is unchanged. Thickening action is also unchanged.

Detailed Description of the Invention

The present invention will be further understood with reference to the following detailed embodiments thereof.

Example 1

Various hydrophobic, pyrogenically produced silicas are investigated, wherein different compaction states are compared.

The following definitions apply:

bulk = pulverulent, unmodified silica

CF = silica compacted with a Carter filter

VV 60 = silica compacted to a tamped density of approx. 60 g/l

VV 90 = silica compacted to a tamped density of approx. 90 g/l

Aerosil grades R 202, US 202, US 204, R 812, R 812S and R 805 are investigated. The results are shown in Table 1.

As evaluated by the Corning Glass methanol wettability method, the degree of compaction has virtually no appreciable influence on hydrophobicity. Viscosity also exhibits no clear systematic dependency upon tamped density. Especially for R 812, dispersibility improves with increasing density. R 812 S, which contains more SiOH groups than R 812, exhibits the above phenomenon less markedly.

US 202 and US 204 have very comparable rheological properties and are inferior to AEROSIL R 202.

Surprisingly, the compacted variants, in particular of R 812, R 202 and US 202/4, exhibit an incorporation time reduced by up to half. The compacted silicas moreover exhibit reduced dusting.

PA	Test method	444701 AER 202 CF	444702 AER 202 CF	444703 AER 202 VV60	444704 AER 202 VV90	444705 AER 805 bulk	444706 AER 805 CF	444707 AER 805 VV60	444708 AER 805 VV90	444709 AER 812 bulk	444710 AER 812 CF	444711 AER 812 VV60	
0330	Viscosity, epoxy before cure	459	456	382	430	190	181	185	178				
0335	Viscosity, epoxy after cure	544	547	494	528	42	417	39	43				
0340	Thickening action									11.7	13.3	11.5	
0410	Grind meter value									127	102	92	
0420	Methanol wettability												
0701	Tamped density	45	50	51	75	44	62	55	68	45	44	50	
0920	Agglomerate strength		11	20	18		15	15	20				
0930	Hunh[sic] sieve oversize	0	24	4	27	0	36	7	19	0	0		
0955	Effectiveness	258	274	203	266	235	260	236	258	166	185	169	
0965	Effectiveness (UT)	280	290	226	295	271	284	270	288	197	213	209	
0975	Settling (effectiveness)	15	15	15	8	10	15	10	5	13	15	8	

PA	Test method	444712 AER 812 VV90	444713 AER 812S bulk	444714 AER 812S CF	444715 AER 812S VV60	444716 AER 812S VV90	444717 US202 bulk	444718 US202 CF	444719 US202 VV60	444720 US204 bulk	444721 US204 CF	444722 US204 VV60	444723 US204 VV90
0330	Viscosity, epoxy before cure						350.4	377.6	380.8	379.2	350.4	358.4	368
0335	Viscosity, epoxy after cure						50.7	45.9	45.3	49.9	47	52.6	50.7
0340	Thickening action	11.1	17.3	17.3	18.2	17							
0410	Grind meter value	77	93	110	110	100							
0120	Methanol wettability												
0701	Tamped density	73	49	50	58	75	39	50	67	44	45	57	71
0920	Agglomerate strength	22				28		10	15			16	23
0930	Hardt[sic] sieve oversize	12	0	0	0	4	0	27	36	0	0	3	20
0955	Effectiveness	159	168	169	187	209	320	304	320	186	193	192	201
0965	Effectiveness (UT)	225	201	200	216	235	336	327	346	223	225	225	230
0975	Settling (effectiveness)	5	8	8	3	0	15	10	3	10	10	10	10

Example 2

Investigation of the influence of higher compaction on applicational properties

		AE R 812, uncompacted UB 3847-1 10 kg sack	AE R 812, compacted RHE UB 3847-2 (4) 15 kg sack	AE R 812, compacted RHE UB 3847-3 (5) 20 kg sack	AE R 812 RHE specific.
Tamped density (DIN ISO 787/11)	g/l	50	87	106	approx. 50
Effectiveness, ethanol (0955)		184	214	209	216 1)
Effectiveness (UT), ethanol (0965)		218	260	290	236 1)
Settling (effectiveness, high-speed mixer)	vol.%	10	1	1	1)

1. Determined on standard sample (UB 3391)

RHE in the above table indicates the Rheinfelden plant located in Germany.

Rheological testing:

Polymer: Araldit M (biphenol-1-expoxy resin by Ciba-Geogy, in the form of clear yellow liquid).

Thixotroping agent: R 202 and R 812 Additive: -

Sample A R 812 10 kg 2-10123

Sample production date: 24.02.1994 Spindle: 5

Storage time in days	5 rpm [mPa*s]	50 rpm [mPa*s]	T.I.
0	16600 80-85 µ	4460	3.72

Sample A R 812 15 kg 1.0/8 min

Sample production date: 24.02.1994 Spindle: 5

Storage time in days	5 rpm [mPa*s]	50 rpm [mPa*s]	T.I.
0	15100 50-60 µ	4060	3.72

Sample A R 812 20 kg 0.6/14 min

Sample production date: 24.02.1994 Spindle: 5

Storage time in days	5 rpm [mPa*s]	50 rpm [mPa*s]	T.I.
0	15100 50-60 µ	4020	3.73

Compaction may amount to a type of predispersion. Accordingly, effectiveness values rise with tamped density, i.e. the particles effectively present in the ethanol dispersion become smaller and

the compacted samples exhibit distinctly less settling. Any suitable organic solvent can be used to form the dispersion.

The compacted samples accordingly have a more favourable Grindometer value in Araldit. However, since the larger particles have a decisive influence on thickening action, the property declines slightly on compaction.

It may be seen from the graph of effectiveness values that, while the highly compacted AEROSIL R 812 sample may indeed still be broken up with the Ultra-Turrax mixer (0965), it can no longer be broken up with the high speed mixer (0955). Due to the smaller surface area of AEROSIL R 202 (and to the consequently theoretically improved dispersibility), this phenomenon hardly occurs with AEROSIL R 202.

As compaction rises, the particles effectively present in an ethanol dispersion thus become smaller and 90° angle scattering rises due to Rayleigh scattering. Total scattering (over all angles), however, falls and the samples become distinctly more transparent on visual inspection. as is also substantiated by the UV transmission spectra.

Compaction has no influence on hydrophobicity, which in each case substantially corresponds to that of the standard sample.

Example 3

Investigation of the influence of higher compaction on applicational properties.

		AE R 812, uncompacted UB 3848-1 2-02024 10 kg sack	AE R 202, compacted RHE UB 3848-2 2-01024-(2) 15 kg sack	AE R 202, compacted RHE UB 3848-3 2-01024-(3) 20 kg sack	AE R 202 RHE specific.
Tamped density (DIN ISO 787/11)	g/l	51	93	119	approx. 60 3)
Effectiveness, ethanol (0955)		319	334	336	334 1)
Effectiveness (UT), ethanol (0965)		346	365	373	339 1)
Settling (effectiveness, high-speed mixer)	vol.%	10	5	1	

1) Determined on standard sample (UB 3391)

3) Guide value

The compacted AEROSIL R 202 samples behave in a similar manner to the compacted AEROSIL R 812 samples.

Reference is thus made to Example 2 with regard to the discussion.

The parameter of "effectiveness" reported in the tables herein relates to the high degree of fineness of the particle. This is therefore an indicator of high transparency and good stability of the resulting dispersions.

Further variations and modifications of the foregoing will be apparent to those skilled in the art and are intended to be encompassed by the claims appended hereto.

German priority application filed December 22, 2000 199 61 933.6 is relied on and incorporated herein by reference.